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IS 12869-1 (1989): Methods for Determination of Viscosity and Viscometric Fixed Points of Glass, Part 1: Determination of Viscosity by Fibre Elongation Method [CHD 10: Glassware]



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“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**METHODS FOR DETERMINATION OF
VISCOSITY AND VISCOMETRIC FIXED
POINTS OF GLASS**

PART 1 DETERMINATION OF VISCOSITY BY FIBRE ELONGATION METHOD

भारतीय मानक

काँच की श्यानता और श्यानतामितीय बिन्दु ज्ञात करने की पद्धति

भाग 1 रेखा द्रैर्घीकरण पद्धति द्वारा श्यानता ज्ञात करना

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FOREWORD

This Indian Standard (Part 1) was adopted by the Bureau of Indian Standards on 28 November 1989 after the draft finalized by the Glassware Sectional Committee had been approved by the Chemical Division Council.

The quality and the type of glass is very much dependent upon the relationship of viscosity of glass with temperature change. Important information of glass technology regarding the temperature required to produce a homogeneous melt for production of glass and the temperature range of formation of glass can be collected from this relationship. This standard, therefore, also provides guidance for determining viscosity/temperature relationship.

This standard is based on ISO 7884-3 'Glass — Viscosity and viscometric fixed points — Part 3 Determination of viscosity by fibre elongation viscometer'. The departure from ISO document is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS FOR DETERMINATION OF VISCOSITY AND VISCOMETRIC FIXED POINTS OF GLASS

PART 1 DETERMINATION OF VISCOSITY BY FIBRE ELONGATION METHOD

1 SCOPE

1.1 This standard (Part 1) prescribes the method for determining the viscosity of glass by measuring the elongation of a glass fibre under a defined uniaxial stress. In addition, the viscosity/temperature relationship (see Annex A) and the dependence of viscosity on thermal history can be determined.

NOTE — The viscous flow by the glass fibre is assumed to be Newtonian. The glass is presumed to be homogeneous and isotropic and the effect of gravity on fibre is neglected. When the fibre shape and cross-sectional tolerance conform to sample specifications given in this standard, all parts of the fibre can be presumed to be subjected to the same mechanical stress leading to a pure elongation.

1.2 The knowledge of equilibrium viscosities in the low temperature range with their dependence on temperature and thermal history will be extremely useful in predicting glass properties and behaviour.

2 REFERENCES

2.1 The following Indian Standard is a necessary adjunct to this standard:

IS No.	Title
7358 : 1984	Thermocouples

3 FIELD OF APPLICATION

3.1 The method is applicable mainly to glass viscosity range from 10^8 to 10^{13} dPa.s* with possibilities for extending the range to 10^{14-5} dPa.s with special devices. This corresponds to a temperature range of 800°C to 400°C for normal commercial glass compositions being produced in bulk quantities.

NOTE — In the viscosity ranges above 10^{13} dPa.s the thermal equilibrium of the glass structure takes considerable time to readjust following a temperature change. If equilibrium measurements are envisaged, sufficient time must be allowed following the change of temperature for thermal equilibrium to be re-established before the elongation measurement is undertaken. Alternatively a dynamic measurement can be done involving measurements made during a well defined time/temperature programme. These measurements will correspond to a dynamical, non-equilibrium value for the viscosity η_{neq} indicated by which may nevertheless be consistent and reproducible under defined time temperature programme. The symbol without index denotes always equilibrium viscosity.

$$*1 \text{ dPa.s} = 1 \frac{\text{dN.s}}{\text{m}^2} = 1 \text{ P}$$

where dPa.s is deci pascal second, $\frac{\text{dN.s}}{\text{m}^2}$ is deci Newton second per meter square and P = poise.

4 TERMINOLOGY

4.1 Viscosity

The property of resistance to flow under stress. In the case of Newtonian behaviour, the rate of deformation is proportional to the stress. The preferred unit for the viscosity of glass is the sub-unit decipascal second (dPa.s)*.

4.2 Ranges of Viscosity

With respect to practical application, three ranges of viscosity measurement can be distinguished:

- a) melting range — up to 10^3 dPa.s
- b) working range — from 10^3 to 10^8 dPa.s
- c) annealing range — from 10^{13} to 10^{15} dPa.s

4.3 Viscometric Fixed Points

It is convenient to prefer the following four temperatures to characterize the viscosity/temperature behaviour of a glass.

NOTE — The expression 'fixed point' does not denote any relation to thermodynamical fixed points.

4.3.1 Working Point T_{11}

The temperature corresponding to a viscosity (η_{11}) of 10^4 dPa.s.

4.3.2 Softening Point T_{12}

The temperature above which the glass is capable of most forming operations. The softening point is also called Littleton temperature. The corresponding viscosity (η_{12}) is estimated by the following equation:

$$\eta_{12} = 2.1 \times 10^7 (\rho - \sigma/520)$$

where

ρ = density of glass in grams per cubic centimetre;

σ = surface tension in millinewtons per metre.

For $\rho = 2.5 \text{ g/cm}^3$ and $\sigma = 300 \text{ mN/m}$ the viscosity is $\eta_{12} = 10^{7.6} \text{ dPa.s}$

$$*1 \text{ dPa.s} = 1 \frac{\text{dN.s}}{\text{m}^2} = 1 \text{ P}$$

(P is the symbol for poise).

4.3.3 Annealing Point T_{13}

The temperature at which internal stresses in a glass are substantially relieved in a matter of minutes. The temperature at the annealing point corresponding to a viscosity (η_{13}) from 10^{13} to $10^{13.2}$ dPa.s.

4.3.3.1 Annealing range

The range of temperature in which stresses in glass articles can be relieved at a commercially desirable rate.

4.3.4 Strain Point T_{14}

The temperature at which internal stresses in a glass are substantially relieved in a matter of hours. The temperature at the strain point corresponding to a viscosity (η_{14}) from $10^{14.5}$ to $10^{14.7}$ dPa.s.

4.4 Fibre

The fibre is the cylindrical part of the specimen with constant cross-section $S = \frac{\pi d^2}{4}$ over its length where d is the uniform diameter of the fibre drawn from molten glass.

4.5 Fibre Ball

A fibre having two ends in the shape of spherical balls and prepared by fusing each end of the fibre on burner flame into spherical ball shape enabling the suspension of the fibre vertically, the upper ball resting on a suitable slot in a fixed support while a loading device with a suitable slot hangs supported on the lower ball.

4.6 Load

It consists of all parts of the apparatus on which gravity acts to produce a force on the lower end of the fibre.

4.7 Loading Pieces

It is a device for suspending the loading pieces (corresponding to a range of loads) below the specimen in an axial direction including the lower extension rod.

4.8 Dead Weight

It stems from the part of the fibre below the cross-section under consideration. The force due to gravity varies linearly from zero at the lower end to the upper end of the fibre.

4.9 Surface Tension Effect

It acts along the length of fibre by shortening the length. It, therefore, opposes the gravitational force due to the load.

5 PRINCIPLE

5.1 Basic Relation

Consider the force F_0 of the load acting on a specimen prepared from a glass with Newtonian

flow behaviour and with no defects. With surface tension and the dead weight of the fibre being neglected, equation (1) gives a good approximation for the extensional flow rate $\frac{dl}{dt}$ at constant volume V .

$$\frac{dl}{dt} = \frac{1}{3} \times \frac{1}{\eta} F_0 \times \frac{l^2}{V} \quad \dots \quad (1)$$

where

η = viscosity in deci pascal second (dPa.s),

V = volume of the fibre,

$F_0 = mg$ where m is the total mass contributing to F_0 and g is the acceleration due to gravity, and

l = length of the fibre.

From the measurement of length as a function of time for a given load, a straight line plot of $\frac{dl}{dt}$ versus l^2 , can be drawn.

From the slope of the straight line η can be calculated. In practice, the initial length l_0 of the fibre is estimated independently and the elongation (increases in length) is measured by observing a fixed mark on the loading device by using a travelling microscope/cathetometer provided with a vernier scale or an eye piece micrometer.

5.2 Order of Magnitudes of Elongation

For an elongation from l_1 to l_2 ; ($l_2 - l_1 = \delta l$) during the time Δt an expression is obtained by integrating and rewriting (1) as

$$\eta = \frac{g}{3} \times m \times \frac{\Delta t}{V} \times \frac{l_2 \times l_1}{l_2 - l_1} \quad \dots \quad (2)$$

$$= 32\,700 \times m \times \Delta t \times \frac{l_1}{V} \times \frac{(l_1 + \delta l/l_1)}{\delta l/l_1} \quad \dots \quad (3)$$

where $l_1, l_2, \delta l$; are in millimeters, V is in cubic millimeters, m is in g, Δt is in seconds and η is in dPa.s.

For a given diameter of the fibre at the start of experiment d (presuming a constant volume $V = \frac{\pi d^2}{4} \times l_1$) it is possible to calculate the time

interval for extension ratio $\frac{\delta l}{l_1}$ of say 0.02 at different loads for a given viscosity. These values of t_s are tabulated (see Table 1) for an initial diameter of the fibre at $d = 0.737$ mm and $\frac{\delta l}{l_1} = 0.02$.

From the Table 1, it is clear that for the same extension ratio, for a range of temperatures for which the viscosity increases by six orders of magnitude (10^8 to 10^{14}) the extension ratios have to be correspondingly reduced by almost similar orders of magnitude in order to keep reasonably convenient time intervals. This implies increasing the accuracy of measurement of δl by similar orders of magnitude.

**Table 1 Time Interval t_s for an Extension $\delta l/l_1 = 0.02$ Under a Load of Mass m
(As Corrected for Dead Weight and Surface Tension), for a Fibre of
Initial Diameter $d_1 = 0.737$ mm
(Clauses 5.2 and 6.4.2)**

Viscosity dPa s	Time Interval t_s in Seconds for Load m				
	1 g	4 g	16 g	64 g	256 g
(1)	(2)	(3)	(4)	(5)	(6)
10^8	25.6	—	—	—	—
10^9	256	64	—	—	—
10^{10}	2 560	640	160	40	—
10^{11}	25 600	6 400	1 600	400	100
10^{12}	256 000	64 000	16 000	4 000	1 000
10^{13}	—	—	160 000	40 000	10 000
10^{14}	—	—	—	—	100 000

6 APPARATUS

6.1 Viscometer Furnace

A vertical mounted muffle furnace with both ends open, being electrically heated and with a temperature range of up to 900°C is suitable. It must have provisions for measuring the temperature and its distribution along the length of the fibre specimen using thermo-couples. The temperature gradient in the region of the specimen shall not exceed 0.2 °C/cm.

6.1.1 The temperature control must be effected so as to ensure a consistency of temperature in respect of time at any point within the working space to within $\pm 1^\circ\text{C}$ or better. Temperature variations must be effected with consistent and repeatable linear programmes.

6.2 Temperature Measuring and Indicating Instruments

6.2.1 Alumina insulated platinum — 10 percent rhodium/platinum (Type S of IS 7358 : 1984) thermocouples or nickel-chromium/nickel (Type K of IS 7358 : 1984) thermocouples shall be employed. They shall have low thermal inertia with wire diameters not more than 0.5 mm and sufficient immersion length of the hot junction inside the space where temperature is measured.

6.2.2 Control thermocouple hot junctions should be located as close as possible to the furnace heating elements to ensure fast response between any temperature variation and the corrective action by the controls. The hot junctions of the thermocouples for measuring specimen temperatures should be close to specimens (see A in Fig. 1). The temperature distribution along the length of the fibre shall be monitored by suitable devices, for example:

- a) a nickel block of high thermal mass surrounding the fibre in a cylindrical shape with a number of holes positioned at various points distributed evenly covering the length of the fibre thermocouples being inserted through the holes, and

- b) alternatively, a mobile thermocouple of low thermal mass (such as B in Fig. 1).

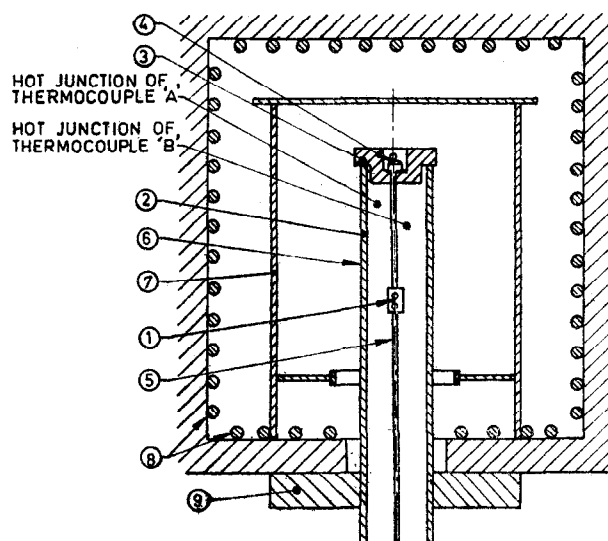
6.2.2.1 The thermocouples shall be calibrated in accordance with the procedure given in IS 7358 : 1984.

6.2.3 The electrical outputs of the thermocouples shall be measured potentiometrically, drawing nil currents or by a high resistance electronic amplifiers having a sensitivity of 1 micro volt for type S (IS 7358 : 1984) and of 4 micro volt for Type K (IS 7358 : 1984) thermocouples. The cold junctions of the thermocouples shall be maintained at 0°C throughout; alternatively the measuring instrument shall be provided with automatic cold junction compensation device.

6.3 Hanging Device for Specimen and Load

The fibre is freely suspended from a connecting device by placing the upper ball of fibre in the lower groove of the device (Fig. 2 and 3). A fused silica rod with ball ends is used as an upper extension for the suspension, the lower ball of this rod supports the connecting device at its upper groove. The upper ball of the silica rod rests centrally on a suitable groove of metal plate of ceramic plate placed on the top of the muffle tube. The loading linkage consists of a fused silica rod of about 1 mm diameter with fused balls on both ends. The upper ball rests on the lower groove of the connector which is suspended from the fibre. A similar connector hanging from the lower end of this rod and extending outside the furnace, can be used for loading.

6.3.1 It is absolutely essential that the extension rods and the fibre hang vertically, coaxially and centrally with reference to the furnace muffle tube. While measuring viscosities in the range of 10^8 to 10^9 dPa.s, the loading may entirely consist of only the lower extension rod of fused silica of approximately 0.5 mm diameter and a total mass of about 1 g. No additional loading is necessary.



- | | |
|--|---|
| 1 Connector for the lower ball of the test specimen and the loading device (for connectors offering different magnitudes of load, see Figures 3 and 4) | 5 Loading device (only the rod is shown) |
| 2 Test specimen | 6 Support-stand : tube of fused silica |
| 3 Support plate | 7 Screening between working space and heaters |
| 4 Upper ball of the specimen | 8 Chamber furnace with heaters |
| | 9 Cover ring below the furnace (fixed at the silica tube) |

FIG. 1 FIBRE ELONGATION METHOD : EXAMPLE OF A TESTING DEVICE (SCHEMATIC)

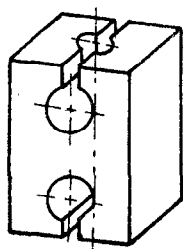


FIG. 2 CONNECTOR BLOCK (METAL) BORED TO ACT AS HANGING DEVICE

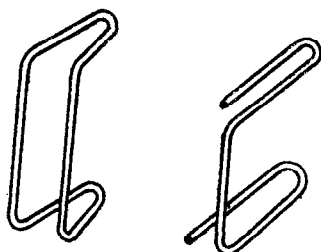


FIG. 3 CONNECTOR HOOKS (WIRE) TO ACT AS HANGING DEVICE

6.4 Loading Pieces

While measuring medium viscosity ranges 10^{10} to 10^{11} , the total load on fibre should be around 4 g. The lower silica extension rod should have a diameter around 1 mm.

6.4.1 For higher viscosities heavier loads will be necessary and the lower silica extension rod must have a larger diameter.

6.4.2 A set of brass loading pieces with masses determined to an accuracy of 0.01 g may be used. The pieces shall be shaped suitably, to be hung symmetrically from a hook which can be connected to the lower extension fused silica rod by the connecting device. An idea of the masses for the brass pieces can be had from Table 1.

6.5 Measurement of Elongation

6.5.1 Use of Optical Devices

A mark is made on the load carrying device outside the furnace. A cathetometer or travelling microscope fitted with scale and vernier can be used. The vernier must have a least count of at least 0.02 mm. The error arising from graduation of the scale and vernier shall be such as to ensure that the error in measurement of δl does not exceed 0.01 mm. The instrument (travelling microscope or cathetometer) shall be rigidly locked in position during any one series of determinations. It shall be conveniently positioned in advance so that the maximum extension of the fibre will still be within the range of its observation.

6.5.2 Measurement of the Initial Length l_1

The length l_0 of the fibre between the two ball shaped ends is measured at the room temperature using a travelling microscope or cathetometer. The fibre is then suspended in position with the extension rods, connectors and loading device in the furnace at room temperature. The measuring instrument is then focussed on to the moving

mark and the scale readings noted. At any subsequent measurements at different temperatures and loads, increases in the length of the fibre can be measured by changes in scale readings on subsequent settings of the optical device. Calibration for initial fibre lengths l_1 at any temperatures is possible using a pure platinum fibre (say T.C., wire of 0.5 mm) of similar dimensions and with fused spherical ball ends. The length of platinum fibre can be measured accurately and its coefficient of expansion is also known. At any temperature a reading on the moving marker with the platinum fibre in position in place of the glass fibre will correspond to a known length of the fibre.

6.5.3 Use of Linear Variable Differential Transformers (LVDT)

Instead of an optical device for the measurement of δl , an extension rod from the loading device can be coupled to an LVDT (linear variable differential transformer) comprising a core and coil separated from each other. The relative movement between the core and the coil measures the displacement. This displacement is displayed or recorded as an electrical output. The possibility of adjustable amplifications for the signal is an added advantage enabling a wide range of sensitivities without disturbing the mechanical settings. LVDT's with the required sensitivities and accuracies are easily available.

6.5.4 Blast flame burner, two rods of stainless steel, ceramics or hard glass for flame working.

6.5.5 Electrically heated crucible glass melting furnace with arrangement for fibre drawing, such as water cooled copper coils; pull rod of platinum, or stainless steel of fused silica.

6.5.6 Timer Devices

Timer devices are necessary covering a range of 20 second to 2000 seconds with a least count of 0.1 second. The accuracy of the time should be ensured to better than 0.2 percent.

6.5.7 Fibre Diameter

A micrometer screw gauge with a least count of at least 0.005 mm is required.

7 SAMPLE PREPARATION

7.1 Preparation of Fibre by Flame Working

This method can be used only when the glass is stable with heat treatment without any tendency for devitrification or phase separation. A piece of the glass about 2 to 3 cm³ in volume is fused in the flame by holding it between the rods (of platinum, ceramics or harder glasses) and turning it around. When it is sufficiently soft it is suddenly withdrawn from the flame and drawn apart using the rods. The operation shall be completed quickly to prevent loss of volatiles in the flame working. Skill is required in this operation with respect to:

- a) choice of temperature (as judged by the softness of the work pieces) at which the drawing apart is undertaken,

- b) force and speed of drawing to ensure fibre of particular diameter,
- c) uniformity of drawing, and
- d) stopping of drawing at the right time.

7.2 Preparation of Fibre from the Melt

About 100 g of the glass is melted in a platinum crucible in the electrically heated furnace. The glass can be fed in the form of cullet but the grains should not be finer than 3 mm to avoid bubbles. When the glass is completely melted and free from bubbles a pull rod of platinum, ceramic or fused silica is dipped into the melt. The rod is pulled up quickly through a water cooled copper coil to the desired length. A similar degree of skill in the drawing is required as indicated for flame working (6.1). A mechanised device for pulling ensures repeatable and well controlled drawing speeds.

7.3 Specimen Preparation for Elongation Experiment

About 50 to 100 mm length of the fibre will be required for the elongation measurement. A length of about 20 to 30 mm longer than the desired length of fibre is taken and the portion of uniform round diameter is chosen. By flame working the two ends are made into spherical balls of about 2 mm. The balls are properly centred on the fibre axis.

7.3.1 After the fibre has cooled completely the length of the fibre is measured between the two balls, accurately to 0.2 mm. The diameter can be between 0.6 to 0.8 mm for most measurements. At the high temperature (low viscosities of 10⁸ to 10⁹ dPa.s) the diameter could better be up to 1 mm. A number of measurement of the diameter is made equally distributed along the length of the fibre, and at each point in two directions at right angles to each other. The maximum and minimum diameters shall not differ by more than 2.5 percent of the mean diameter. The arithmetic mean value of the diameter shall be used for calculating the fibre volume V .

7.3.2 The upper end of the fibre (when suspended for elongation measurements) shall be chosen to have the larger diameter ball.

7.3.3 The test specimen shall be free from scratches, striae, devitrification, bubbles or other inhomogeneities. There shall be no neck formation of fibre at either end close to the balls.

7.4 Etching of the Test Specimen

An etching solution shall be prepared with 2 mol/litre aqueous solution of hydrochloric, sulphuric or nitric acid containing 3 percent of hydrofluoric acid. The glass fibre is placed in this solution at room temperature for 0.5 to 1 minute. The fibre is taken out, rinsed with distilled water and dried.

8 MEASUREMENT PROCEDURE

8.1 Calibration

The fibre elongation method based on equation (1) is an absolute method for determining the viscosity calibration involved only for thermocouples. The thermocouples must be calibrated against certified standard thermocouples at regular intervals.

8.2 Determination of Viscosity at Constant Temperatures

Heat the furnace to the lowest point in the temperature range within which the measurement is desired. When the furnace temperature is stable introduce the fibre with the support and extension rods. Wait for a few minutes for the temperature equilibrium to be re-established. Focus optical instrument on the moving marker and note the scale reading. In case LVDT is used, zero the chart on recorder at this point or note the reading if visual indicator only is used.

8.2.1 The length l_1 of the fibre in this position is known (6.5.2).

8.2.2 Load pieces may be added. The cathetometer/travelling microscope is focussed on the mark and the timer started. In the case of LVDT the recorder is zeroed and the chart started simultaneously. If visual recording of LVDT output is used, a timer is started simultaneously with zeroing of LVDT output. The vernier of travelling microscope/cathetometer is moved by a known increment of length and the time when the mark crosses the reference line in the eyepiece is recorded. The scale is then shifted to the next selected increment. At least six positions of lengths and the corresponding times are recorded using travelling microscope/cathetometer or LVDT for a given temperature.

8.2.3 Remove the loads and heat the furnace to the next higher temperature point. After stabilisation of temperature add the load pieces and repeat measurements.

8.2.4 At high temperatures corresponding to viscosities 10^9 to 10^8 dPa.s, no load pieces will be required. The dead load is sufficient for the measurement.

8.2.5 Smaller elongations with higher sensitivities for measurement of δl are preferred and the elongation during and one test shall not exceed 2 percent of the starting length.

8.2.6 At high viscosities 10^{11} dPa.s and above delayed elastic effects become important and sufficient time shall be allowed to elapse after the addition of the load, before the readings are taken.

8.3 Finishing of Test Procedure

The test shall be completed before any one fibre elongates by 10 percent of l_1 . Remove the test fibre from the furnace and allow it to cool to room temperature. Inspect the fibre for devitrification, striae, scratches and neckings. Again determine the length of the fibre as well as the mean diameter and note if the cross-section has

deviated from circular geometry. The difference between the maximum and minimum diameter shall not exceed 4 percent of the mean.

9 EXPRESSION OF RESULTS

9.1 Corrections for Load

When the loading mass is 4 g or less, correction must be made to include the mass of the fibre and surface tension shall also be accounted for. These are taken care of by substituting for m_1 , and m_2 given by:

$$m_2 = m_1 + f\rho - f\sigma$$

where

$$f\rho = 0.0005 \times \rho \times V$$

$$f\sigma = 0.00018 \times \sigma \times \sqrt{V}/l_1$$

ρ = density of the glass in g/cc

σ = surface tension of the glass in milli Newtons/meter

when σ is unknown, an idea of its effect can be had using the value $\sigma = 330$ mN/m.

9.2 Evaluation of Measurements

9.2.1 A plot of length of fibre against time can be made from which, $\frac{dl}{dt}$ at different l can be evaluated. A plot of $\frac{dl}{dt}$ against l^2 must yield a straight line plot [see equation (1) 5.1] from the slope of which η can be calculated. At least six points shall be plotted for straight line.

9.2.2 The expression given in equation (3) 5.2 can also be used with δl evaluated for a given l_1 and interval of time Δt . At least six different readings of δl shall be used to get average value for η .

9.2.3 Representation of Results

The viscosity values at the different temperatures may be plotted with $\log \eta$ on the ordinate and temperature on the abscissa. This relationship between η and T is governed by the Vogel Fulcher Tammar (VFT) equation (see Annex A)

$\log \eta = A + \frac{B}{T-C}$; This enables interpolation of $\log \eta$ /Temperature values at points other than those at which test were done. The constants A , B , C may be determined from any three sets of η , T values from the $\log \eta$, T curves.

10 TEST REPORT

10.1 The test report shall include:

- reference to this Indian Standard,
- description of sample,
- method of sampling,
- number of test specimens,
- method of preparation of specimen,
- corrections applied,
- plot of $\log$ versus reciprocal of temperature according to VFT equation, and
- any change in glass, observed during and/or after the test.

ANNEX A

(*Clauses 1.1 and 9.2.3*)

VISCOSITY/TEMPERATURE RELATIONSHIP

A-1 THE VFT-EQUATION

A-1.1 For the purpose of interpolation the viscosity/temperature relationship is conveniently described by the equation of Vogel Fulcher and Tamman (the VFT-equation) :

$$\log \eta = A + \frac{B}{T-C} \quad \dots\dots\dots(1)$$

The value of the dynamic viscosity η_{neg} shall be inserted in decipascal seconds (dPa.s), and the temperature T in degrees Celsius (°C).

The constants A , B and C are characteristics for the glass under test.

NOTE — The order of magnitude of the constants for technical glasses is as follows:

$A = 10^\circ$ (decadic logarithm of viscosity in decipascal seconds)

$B = 10^3 \text{ }^\circ\text{C}$

$C = 10^2 \text{ }^\circ\text{C}$

A-2 DETERMINATION OF THE CONSTANTS

A-2.1 Three pairs of measured temperatures and equilibrium viscosities is equal to 1, 2, 3, covering the range of the measurement and separated sufficiently from each other, are chosen to calculate the constants of the VFT-equation by means of equations (2) to (4) :

$$C = \frac{(T_2 - T_1)(T_3 - T_1) \times (\log \eta_3 - \log \eta_2)}{(T_2 - T_1)(\log \eta_3 - \log \eta_1) - (T_3 - T_1)(\log \eta_2 - \log \eta_1)} + T_1 \quad \dots\dots\dots(2)$$

$$A = \frac{\log \eta_2 \times (T_2 - C) - \log \eta_1 \times (T_1 - C)}{(T_2 - T_1)} \quad \dots\dots\dots(3)$$

$$B = (T_1 - C) \log \eta_1 - A \quad \dots\dots\dots(4)$$

As a proof of the evaluation, calculate the constant B once more, using equilibrium viscosity is equal to 2 or 3, respectively.

A-3 TEMPERATURE COEFFICIENT OF VISCOSITY

A-3.1 The temperature coefficient of the viscosity of a glass is defined by equation (5):

$$U_n = \frac{1}{\eta} \times \frac{d\eta}{dT} \quad \dots\dots\dots(5)$$

Using the VFT-equation, the temperature coefficient U_n is given by equation (6) :

$$U_n = \frac{2.303 \times B}{(T - C)^2} \quad \dots\dots\dots(6)$$

A-4 ERROR CHARACTERIZATION

A-4.1 The deviation of a measured point from a fitted temperature/viscosity curve (for example found by regression analysis) can be expressed either by a viscosity difference $\Delta \log \eta$ or by a temperature difference ΔT . These differences are related to one another by equation (7) :

$$\Delta \log \eta = 0.4343 \times U_n \times \Delta T \quad \dots\dots\dots(7)$$

Where U_n is the mean temperature coefficient of viscosity in the corresponding range of temperature.

A-5 VISCOSITY/TEMPERATURE PLOT

The graphical representation of the viscosity/temperature relationship is usually performed by plotting the logarithm of viscosity as ordinate against the linear temperature axis as abscissa.

ANNEX B

(Foreword)

DEPARTURE FROM ISO DOCUMENT

B-1 The departure from ISO document is as follows.

B-2 ELONGATION RATE

B-2.1 While describing the procedure for determination of elongation of the fibre, the ISO document prescribes that after loading the fibre, one should wait 'for nearly constant elongation rate', before proceeding to record elongation as a function of time. It is apparent from the basic expression relating elongation rate to at a given temperature that the elongation rate is directly proportional to l^2 . Hence elongation rate will never be constant. This has been taken into account.

B-3 INITIAL LENGTH OF FIBRE

B-3.1 A cathetometer or telemicroscope focussed on a well defined mark on the extension rod or loading device for the fibre enables measurement of increments in length of the fibre. However, the initial length of the fibre inside the furnace at the test temperature must be known for correlating the elongation rate to the viscosity at the temperature. This is normally taken care of by measuring the length of fibre at room temperature using vernier calipers or travelling microscopes. The measuring device is focussed on the mark after suspending the fibre at room temperature inside the furnace. As the furnace temperature is raised, increments in length are monitored by following the moving mark on the optical measuring device. However, this procedure makes it imperative that the optical measuring instrument must be absolutely stationary with rigid fixing devices through out the measurement procedure for the entire range of temperatures and hence the entire measurement must be completed in a day.

The use of a pure platinum wire (T. C. grade) of about 0.5 mm dia or more will provide a very convenient standard length with reference to which positions of initial readings of optical measuring devices can be related to specific initial lengths in the furnace. The platinum wire must have spherical ends by flame fusion, in an oxy-acetylene flame or electric arc and suspended inside the furnace at any desired temperature in the same manner as the glass fibre. The coefficient of linear thermal expansion of pure platinum (T. C. grade) is well defined and thus an accurate initial length position can be known corresponding to the reading of the optical devices on the standard mark when the platinum wire is suspended at any temperature and even after an interruption in the procedure entailing disturbance of the optical measuring devices. The use of the platinum wire has, therefore, been incorporated.

B-4 MEASUREMENT PROCEDURE

B-4.1 Recording of increment in length as a function of time at any given temperature enables a plot of $\frac{dl}{dt}$ versus l^2 . This must be a straight line yielding an elegant method for calculating η from the slope. This procedure has been incorporated in preference to calculating η from individual values of l_1 , l_2 and the corresponding time interval.

B-4.2 The dynamic methods for measurement involving determination of elongation rates while the furnace temperature is programmed for linear rise and fall has not been included since it is felt that the precision of rate control and exact repeatability of programmes may be very difficult to achieve.

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